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R. E. Roche or George

REPORT NO. 1278

Distillation, Drying and Testing Purity of Hg.

Genaral:

Most Mercury that comes in the laboratory for treatment, contains substances which effect its use as a reagent therefore it is necessary to follow the outline as here-after described, in order that those foreign substances which effect its actions as a reagent may be removed.

Procedure:

1. Initial Wash

Take 25 lbs of impure or contaminated ~~Hg. Place in 1500 or 2000~~ cc container and wash by passing Hot H_2O through the Hg for at least 4 hrs. depending upon the amount of organic or other foreign substances present . Note; (In case oils are present, this time should be increased to 6 hrs.). The hot water which is passed through should be at such a rate as to break the surface tension of the Hg. This is best obtained by allowing the water to pass through a glass tube and released under the surface of the Hg near the bottom of the bottle or container.

2. Mercury and Water Separation

After completing #1, decant the water above the Mercury and remove all possible water which is on surface, by the use of a vacumn useing a trap to collect the water.

3. Removing amalgams and Acid Neturlization

Take the Mercury from No 2 and pass it through a 30 to 40 cm. tube containing 8% HNO_3. Then through a tuve of the same size containing distilled water. These solutions should be changed after passing thro gh 10 lbs of Hg. The rate at which the Hg is passed through these solutions should exceed not 5 lbs per hour.

4. First Drying (Drierrite method)

The Hg from No 3 is passed over a tube containing Drierite. The tube should be form 12 to 14 Ml. in diameter and 30 cm. in lenght. Place glass wool at top and botten of Drierite column so as to insure diffusion of Mercury allowing a greater surface to be exposed. The Mercury level sholuld be kept at 3 to 5 cms. above the upper glass wool mat. The Mercury should not passa from Drierite tube faster than 5 lbs. per hour. The Drierite should be changed after passing 30 lbs of Hg through the column. Note: (The Mercury should not contain more than 0.3 of 1% of water by weight. See No. 7 for test.)

5. Distillation of Mercury

The Mercury from No. 4 is placed in the open top reservoir of still. (See figure 106, page 685, Analytical Chemistary, Treadwell and Hall, Vol2, Quantitative, 9th, English Edition.) Make sure that the distance from the Mercury level in the reservoir is 75 cms, lower than the upper level of the Hg in the distillation flask. (Distillation flask should be ½ full.) The center tube, or condensation tube which is to receive the Mercury vapors, should always extend 1 cm. above the Mercury level in the distillation flask. CAUTION: These distances vary according to the barometric pressure and the amount of heat applied to the distillation flask. At least 5 cms. should be allowed from the top of the Hg in the reservoir, allowing for pressure caused by Hg when heat is applied, and also any changes in barometric reading.

The lower condensation tube, or center tube, should be at least 82 cms in length, measuring from the bottom of the open top Hg reservoir to the

outlet arm of the Hg seal flask, at the lower end of center or condensation tube. (Note: Center or condensation tube should extend to within ½ cm. of the bottom of the Hg seal flask, (Lower flask). To begin distillation, connect vacuum pump to outlet of Hg seal tube (lower receiving flask), and pull the Hg by vaccum to the desired level in the distillation flask, (half full). (1 cm. below the upper end of center or condensation tube). When level is reached add Hg to the open top reservoir bringing the Hg to the calibrated level. Often it is necessary to add small quantities of Hg while vacuum pump is in operation in order to bring Hg to desired level in distillation flask.

Now a small flame is started under distillation flask. (CAUTION: This should be watched constantly, so that the flame is not too high, causing back pressure on the open Hg reservoir, causing the same to overflow.) Under heat the Hg may rise to 1 to 2 cms. inthe reservoir but never higher. The vacuum pump remains in operation until the Hg seal flask is filled to within ½ cm. of the outlet arm. Now close the bottom screw clamp, disconnect the vacuum pump, and allow the distilled Hg to rise to not less than 50 cms. in the condensation or center tube. Then open the screw clamp slowly and place receiving vessel beneath outlet arm of Hg sealing flask and allow Hg to continue.

6. Final Drying (H_2SO_4 Method.)

The Hg from 5 is placed on electric hot plate under hold. Connect to vacuum with trap between vacumm and Hg container. Dry air is introduced to Hg container using a glass tube extending to the bottom of the container, in order to prevent bumping. The air is dried through conc. H_2SO_4 with a trap between the acid and the Hg. This process is allowed to continue for 4 hours at 180 to 200 degrees F. Remove and allow to cool and strain through 4 layers of clean gauze.

7. Test for Moisture Content.

A5 cc. sample of Hg from No. 6 is shaken out with 25 cc. of alcohol which is an aliquote portion of a 500 cc. sample on which a blank has been run, using Fishers reagent. Decant the alcohol, measure and titrate with Fishers reagent. The amount of water present should not exceed 0.01% by weight. (Note: In case Hg contains a higher percentage of moisture return and repeat No.6.

8. Qualitative Test for Metal Radicals.

The principal impurities found in Hgare: Copper, cadmium, zinc, and some times silver and gold. These substances are tested for as outlined in Langes Handbook Of Chemistery, 4th addition, Page 946. Should any of the above be present, repeat No. 3 through 7 inclusive. (Note: Increase HNO_3 used in No. 3 to 10%.

9. Labeling and Accounting of Reagent Mercury

The Hg from No. 8 is labeled (Distilled and Dried Hg.) Moisture content, date prepared, and quanity or weight. The Hg is tightly stoppered and stored, and shall be accounted for as prescribed by chief chemist in charge.

J Geary